Geopolymer Stabilisation of Unfired Earth Masonry Units

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Abstract. Contemporary domestic structures typically use masonry units that are approximately 100mm thick. There is interest in using commercial methods of manufacture to produce earthen bricks that have a similar form factor to conventional masonry The large scale adoption of thin walled unfired earth masonry is dependent on its suitability for use in a load bearing application. High moisture content leading to full saturation, for example as a result of flooding, is a concern for unstablised earth construction, especially as wall thickness reduces. The greatest barrier for earth masonry adoption is the durability of the material when affected by high moisture content. Accidental and intentional wetting of a 100mm thick load bearing unfired earth wall could lead to disproportionate collapse.

The paper presents initial findings from an investigation into the use of geopolymer mechanism as a method of stabilisation. The use of geopolymer mechanism was chosen as a possible method of improving the water resilience. Soil that is used for commercial extruded fired brick production was chosen. The soil was selected as the precursor (source of the required silica and alumina) and this was mixed with various sodium hydroxide and sodium silicate activators.

Specimens were tested both in their dry sate as well as following 24 hours of submersion in water. Compressive strength of cylinders after saturation, was used as an indicator of effective stabilisation. The maximum dry compressive strength achieved was 10.4N/mm^2 with the addition of 5% sodium hydroxide and 20% sodium silicate after curing at 105°C. The most significant contributor to the strength gain was the addition of sodium silicate. Although some of the cylinders were able to be tested under fully saturated conditions the strengths achieved were negligible and insufficient for structural application. The potential for geopolymers as a method of stabilising unfired earth bricks is discussed with respect to the compressive strengths achieved.

Introduction

Earth as a construction material is typically associated with low embodied energy due to the minimal processing required. Along with environmental benefits from using earth, the aesthetic and tactility of the material has led to a resurgence in the use of earth as a construction material that largely died out in the UK in the 19th century (Morton, 2008).

The vernacular use of earth as a construction material, such as rammed earth and cob, has raised the profile of constructing with earth, but greater environmental benefits are likely to be achieved if the material becomes mainstream. The focus of this paper is on extruded earth masonry units. These bricks are produced using the well established extrusion process for fired brick manufacture. Industrial manufacture ensures a consistent high quality, low cost earthen construction unit, that can

be delivered to mass market. Morton (2006) showed that an earthen brick only consumes approximately 14% of the energy required to produce a fired brick.

Within the UK, the conventional wall thickness for a partition or single leaf wall is around 100mm. Modern earthen construction arguably needs to adopt a similar wall thickness as thicker walls lead to increased material use (with associated environmental impacts), reduction of indoor space and a potential increase in cost. Maskell et al. (2013) showed that increasing the wall thickness to approximately 200mm gives the wall construction the same embodied energy as dense concrete block work. It is therefore environmentally and commercially desirable to keep walls as thin as possible.

Heath et al. (2012a) demonstrated the structural feasibility for 100mm thick extruded earth masonry. The compressive strength of the units ranged from 2.8 to 5.1N/mm^2 when measured at ambient temperature and humidity level (Heath et al., 2009). Heath et al. (2009) demonstrated an exponential relationship between compressive strength and moisture content for the extruded earth units. With increasing moisture content the compressive strength decreases. Under full saturation the extruded bricks loose all strength and disintegrate (Maskell et al., 2012).

The reduction in compressive strength under the extreme moisture contents poses the biggest barrier to adoption of earthen masonry in structural applications. Stabilisation is a method by which the inherent physical properties of the soil can be modified. Stabilisation has been used to increase the compressive strength and durability, so that a reduced, but sufficient strength can be retained when fully saturated.

Typically cement and lime have been used to improve the strength and durability properties (Reddy & Gupta, 2006; Walker, 2004). There are associated environmental impacts of using these stabilisers and there is growing interest in using alternative methods (Maskell et al., 2013). Geopolymers are a novel class of binders and could be used as an alternative to cement.

The paper is focused on utilising the underlying mechanism for the production of geopolymers for the stabilisation of extruded earth masonry. The critical design stress for a 100mm thick wall for a typical domestic structure was calculated to be 0.54N/mm^2 (Heath et al., 2012a) and therefore the unit will need to maintain at least this minimal strength. Since durability of the bricks is key to the adoption, a successful stabilisation has been defined by in this study as one sufficient to maintain 1N/mm^2 compressive strength after 24 hours of full submersion. Full submersion may be considered as too harsh and not representative of in-service conditions, but testing under this condition will give confidence as it is equivalent to that for concrete blocks. Saturated performance of earth masonry remains one of the greatest barriers to commercial adoption (Maskell et al., 2012).

Background

The term ‘geopolymer’ can be applied to a range of products with similar underlying chemistry. Amorphous three-dimensional inorganic polymers are formed through the reaction of aluminosilicate precursor, with an alkaline solution activator. The precursors can be sourced from a range of aluminosilicates sources, including natural clays, other natural minerals, calcined clays and industrial by-products. The activators are typically sodium and potassium hydroxides, sodium silicates and sodium carbonates, but others are feasible.

The underlying chemistry and manufacture of geopolymers is described in detail by Duxson et al. (2007) and Davidovits (2008). The majority of the research on clay based geopolymers has used the highly refined aluminosilicate precursor metakaolin. Metakaolin is produced from heating kaolin

followed by grinding to produce a fine powder. Kaolin is a 1:1 clay mineral with one silicate sheet joined to an alumina sheet. Geopolymers using the precursor of uncalcined kaolin or illite is typically a lower strength than metakaolin and are unsuitable for structural concrete (Davidovits, 2008).

Interest in using geopolymer technology for earth construction is due to the mineralogical and chemical makeup of earth. Although obviously variable, cohesive soils that are used for earthen structures have a quantifiable amount of clay mineral content and are inherently a source of aluminosilicates such as kaolin and illite. This means that the natural clay minerals found within the soil can become an essential part of the stabilising mechanism rather than simply being encapsulated by the stabiliser, as is the case of cement. Soil used for the production of fired brick represents an under utilised, unrefined and mixed source of aluminosilicates possibly suitable for geopolymerisation.

The properties of the geopolymer are highly dependent on many factors. These include the chemical makeup of the precursor aluminosilicates, pre-treatment of this precursor (MacKenzie et al., 2007), curing temperature, water content, solids content, alkali type, alkali concentration, pH as well as the ratios of different elements (Khale & Chaudhary, 2007). Khale and Chaudhary (2007) comment that the curing temperature and time were significant with respect to the development of compressive strength.

It has been demonstrated that compressive strength of non-calcined materials, such as kaolinite, can be increased with the addition of calcined materials including slag, fly ash and metakaolin (Xu & Van Deventer, 2002).

Heath et al. (2012b) questions the future availability of slag and fly ash as almost all of the slag is accounted for in Portland cement blends and fly ash production will dwindle in the UK due to a reduction in reliance on coal. Metakaolin can be created by heating kaolin to approximately 750°C, but there are associated detrimental environmental impacts. There is difficulty in quantifying the environmental impacts of metakaolin (Habert et al., 2011), but are expected to be significantly better than the equivalent mass of cement manufacture. Global warming gasses are only released in the grinding and heating of the kaolin, which is less than that of cement, but unlike cement, no carbon dioxide is released directly from the kaolin.

Rahier et al. (2011) investigated the production of geopolymers from untreated kaolinite. Kaolinite, sand and a sodium hydroxide solution were mixed, compressed under a static load of 16N/mm^2 and then heated to 80°C for 24 hours. A maximum compressive strength of over 30N/mm^2 was achieved with 11% of sodium hydroxide (by dry mass of the kaolinite and sand). A 5% addition of sodium hydroxide achieved a compressive strength of over 10N/mm^2, representing significant potential for non-calcined kaolinite.

Soil is made up of a mixture of different minerals and the suitability of different minerals to be used along with kaolinite as precursors was investigated by Xu and Van Deventer (2000). The geopolymerisation of 16 natural minerals were investigated through compressive strength testing. Xu and Van Deventer (2000) comment that the measured compressive strength samples are not representative but indicative due to testing of 20 × 20 × 20mm cube. Strengths ranged from 2.5 to 18.9 N/mm^2 and all minerals were consistently higher in the presence of potassium hydroxide rather than sodium hydroxide. Although the extent of dissolution of the minerals was greater in sodium hydroxide, Xu and Van Deventer (2000) theorised that ion pair theory could explain the difference in compressive strength.

Davidovits (2008) demonstrated the potential of using soil as the precursor with an alkaline activator in a process described as low temperature geopolymeric setting. Varying mechanical properties are achieved with a kaolinite based lateritic soil that is mixed with an alkaline hydroxide that is set under different temperatures. Davidovits (2008) states that a block that is water stable and has a compressive strength of 4 to 6 N/mm^2 can be achieved with the addition of 1 to 2.5% of an alkaline activator. Higher compressive strengths can be achieved with increasing amounts of activator or increasing the temperature.

Considering that significant compressive strength can be achieved by using an alkali activator on clay minerals, then there is potential for mechanism to be utilised to stabilise brick soil. Literature with respect to the compressive strength of these geopolymers mixes when under full saturation conditions is limited. Geopolymerisation of the natural brick soil may achieve the required saturated strength and be suitable for structural applications.

Materials and Methods

Materials. A soil that is used for the manufacture of commercial fired bricks was used. Heath et al. (2009) demonstrated that the soil used for fired bricks was suitable for unfired clay bricks. The physical properties and mineral content of the chosen soil was investigated by Maskell et al. (2012) and represented in Table 1. The soil can be described as a dark brown sandy silt with a plasticity classification of a low plasticity clay (BS5930:1999, 2010). In addition the properties presented by Maskell et al. (2012), the chemical composition of the soil was determined by X-ray Fluorescence analysis. Only the amount of silicon, aluminium and the three main metal oxides are presented along with the Loss On Ignition (LOI).

Table 1: Soil Properties (Maskell et al., 2012)

Properties	%
Physical Properties	
Liquid Limit	24
Plasticity Index	8
Linear Shrinkage	6
Particle Grading	
Sand	33
Silt	46
Clay	16
Mineral Content	
Siderite	2
Hematite	3
Smectite	3
Chlorite	6
Illite	16
Kaolinite	31
Quartz	39
Elemental Composition (wt.)	
SiO_2	57.48
Al_2O_3	17.04
Fe_2O_3	11.36

K_2O	3.72
TiO_2	1.36
LOI	7.4

This research programme was focussed on one particular soil and involved investigating the options for stabilisation of this soil, rather than investigating which soils were suitable geopolymer stabilisation. This does provide a certain limitation as (Xu & Van Deventer, 2002) noted that for uncalcined clays "*It is still not possible to predict quantitatively whether or not a specific Si–Al mineral will indeed be suitable for geopolymerisation*."

The soil has a Si/Al ratio of 3.0 and has a large amount of iron present. The presence of iron is similar to the lateritic soil used by Davidovits (2008) and although the molar ratio is within the range used by Xu and Van Deventer (2000), this is based on the XRF results that include silicon within the quartz. This study is to investigate the soil that is used for the brick manufacture for their suitability to be used for geopolymerisation. There are many different types of soils that are used with varying mineralogical and chemical composition but the focus of this paper is on just this one soil.

The geopolymers activators that were used for this study include sodium hydroxide (NaOH) and sodium silicate with a molar ratio of two ($Na_2O.2SiO_2$). Ranges of quantities for the different additives were added.

Sample Preparation. There has been limited research into the stabilisation of extruded earth which has been attributed to the complexities of laboratory scale manufacturing and testing (Maskell et al., 2012). While Maskell et al. (2012) demonstrated a suitable method to make representative bricks, an alternative method of sample preparation was developed. The small scale extrusion method requires a significant quantity of material to be required, and concerns of setting and instrumental damage meant that a different technique was used.

Initially the sodium hydroxide is dissolved into the required amount of water and the solution allowed to cool. This is then added to the soil and is mixed according to BS EN 196-3:2005 (2005). This method of mixing was chosen as it closely represents the method used for industrial manufacture of bricks. The process of forming following mixing is largely continuous and as such storing the material for any period of time, in this case to allow for further dissolution of the minerals, would be undesirable. Following the mixing the material was compressed into cylindrical moulds that created samples measuring 18 mm in diameter by 37 mm high as shown in Fig. 1.

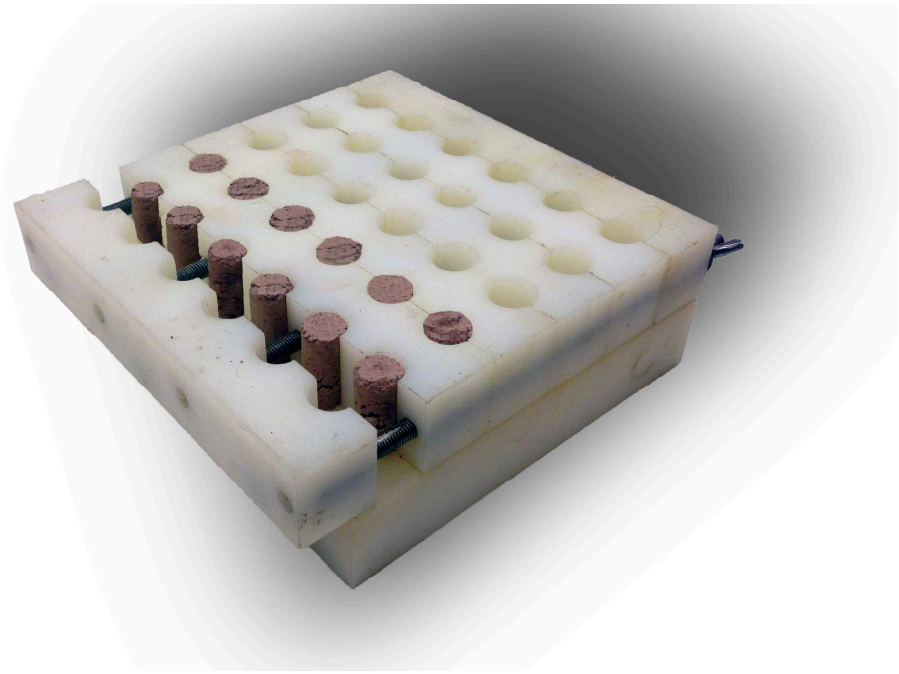

Fig. 1: Cylindrical mould

Optimum Moisture Content. The strength of earthen construction is dependent on the chemical and physical makeup of the soil, the water content, and the density. Maskell et al. (2012) showed that the Optimum Moisture Content (OMC) and maximum dry density of the soil varied depending on compaction or extrusion, with extrusion requiring a higher moisture content. While compaction was used in the sample formation in this study, the ultimate use will to be used within the extrusion process. Therefore, the desired moisture content of the samples is the soil's plastic limit at 16% (Maskell et al., 2012). Any additive to the soil is likely to change the plastic limit and therefore the workability. This initial study added a constant amount of water to all mixtures. The soil in its equilibrium state is at 7% moisture content therefore an additional 9% of water was added.

The density of the samples for any given moisture content is determined by the compactive effort. The same tamping process was used to create all the samples so that approximately the same energy was translated to compaction.

Curing regimes. The curing time was varied to investigate how the stabilisation effect would vary with elevated temperatures and accelerated drying. All the specimens were left drying within the moulds for two days at 21.0°C at 60% relative humidity. After demoulding, half the specimens remained in this condition until testing. The other half were heated to 105°C for two days in an oven after which they were removed and returned to conditions of 21.0°C at 60% relative humidity.

Testing Methods. There are no British or European Standard testing methods for earth masonry. A suitable method of testing extruded earth was discussed by Maskell et al. (2012) with Walker (2004) and Morel et al. (2007) discussing methods of testing CEBs. For this research, compressive strengths were calculated by crushing uncapped specimens and recording the peak compressive load.

Specimens were tested at 28 days and under varying curing conditions. One group of specimens were air dried at 21.0°C at 60% relative humidity, while the other the sample was fully immersed in distilled water for 24 hours prior to testing. These represent testing the specimens 'dry' and 'wet' conditions respectively. Maskell et al. (2012) comments that lack of saturated strength of earth masonry is one of the greatest barrier to its adoption and should be tested even if it is not representative of in-service conditions.

Results and Discussion

There was noticeable variability in the workability of the various mixes. This can be seen with respect to the surface finish as in Fig. 2. In particular the 1% sodium hydroxide and 4% sodium silicate and the 3% sodium hydroxide and 15% sodium silicate were adhering to the sides of the moulds and were unable to be removed without damage.

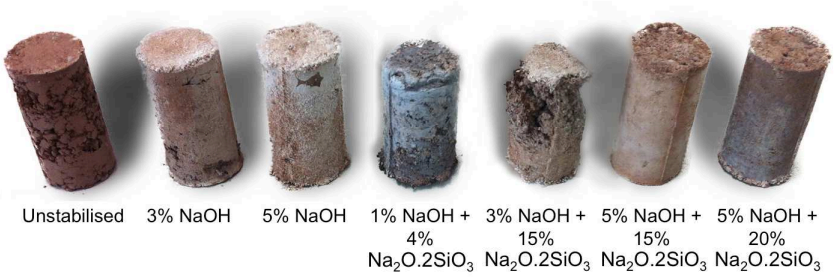

Fig. 2: Earthen cylinder samples

The results are averaged from a sample size of six specimens for each test and are presented in Fig. 3 and Fig. 4. The coefficients of variation for the measured compressive strength tests were all below 20%. The control sample consisted of the same brick clay with no additional stabiliser added and prepared to the same moisture content. Statistical methods of analysis were used with a confidence level of 95%.

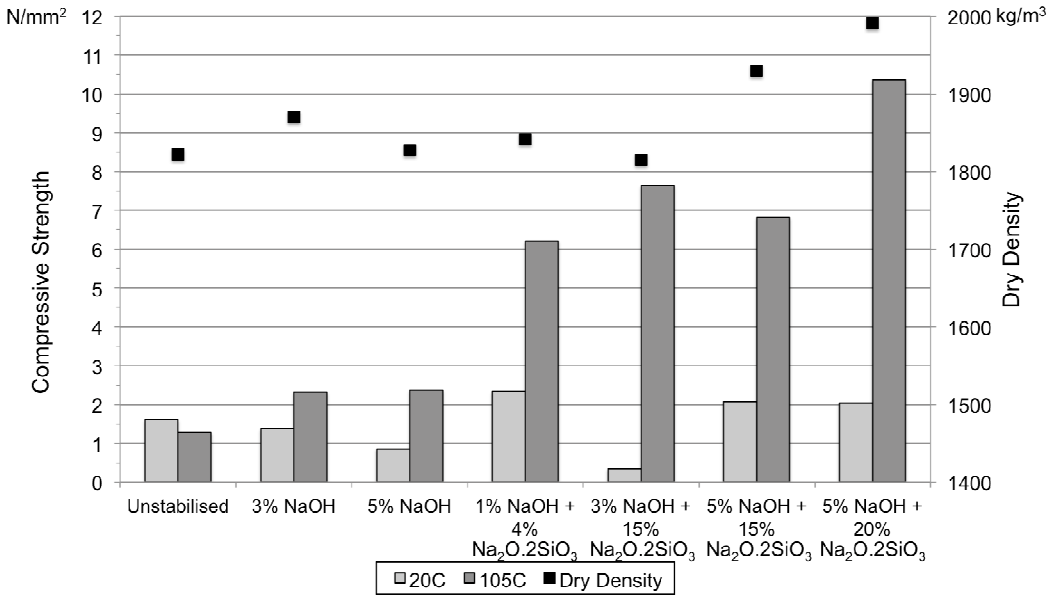

Fig. 3: Dry density and dry compressive strength for different curing temperatures

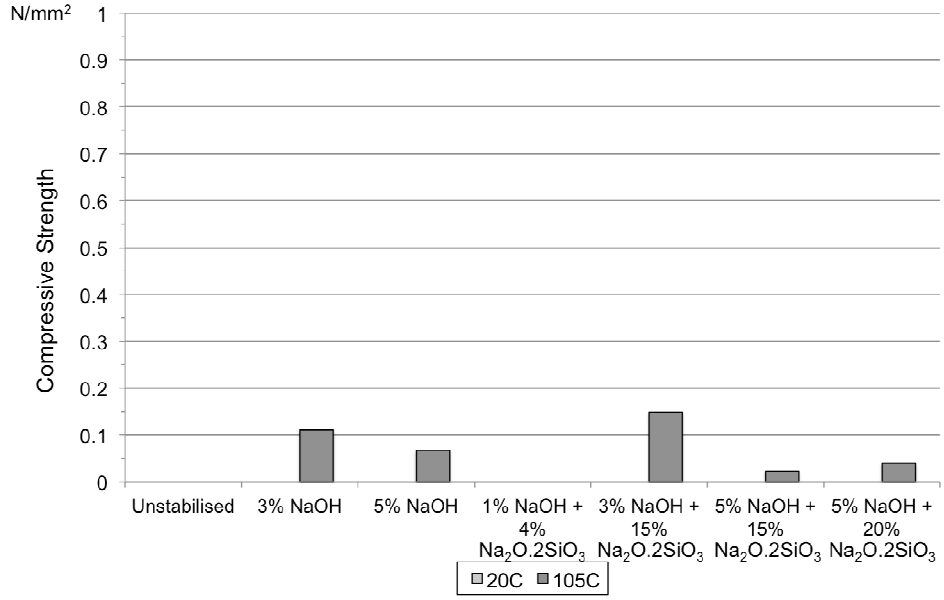

Fig. 4: Wet compressive strength for different curing temperatures

Comparison to earthen bricks. The results for the cylinders can initially be compared to full scale brick results presented by Maskell et al. (2012). The 28 day compressive strengths for full scale scale bricks with a 20°C and 105°C curing regime are 2.75 and 3.05 N/mm^2 respectively. Strengths achieved by testing the cylinders represent 58% and 42% of the measured brick strength. The variation in strength could be attributed to differing factors including density, moisture content and aspect ratio.

The density between the cylinders and full scale bricks are noticeably different. The average dry density of the unstabilised cylinders is 1822kg/m^3 compared to 2157kg/m^3 for the full scale bricks. The method of compaction was unable to represent that achieved by extrusion, which is evident by the voids and surface quality of the cylinders as in Fig.2.

The compressive strength of the earthen material is related exponentially to the moisture content (Heath et al., 2009). The moisture content has been shown to vary between small samples and full scale samples, which can be corrected for (Maskell et al., 2012). This difference in moisture content could be attributed to the rate of drying which would be significantly greater in the small cylinders due to the greater surface area to volume ratio. The average moisture content of the unstabilised cylinders is 2.0 and 1.7% for the 20 and 105°C curing regime respectively, which is significantly lower than the full scale bricks.

The aspect ratio of the cylinders is 2.0 while a bricks aspect ratio is 0.57, which is likely to cause varying platen restrain effects. The effect of platen restraint decreases with increasing distance between the platens. Maskell et al. (2012) demonstrated the effect of apparent compressive strength with varying aspect ratios of bricks. Although the cross sectional areas between the cylinders and bricks differ, the correction factors provided by Maskell et al. (2012) can be used to estimate the effect of aspect ratio. The different aspect ratio accounts for approximately 80% of the variation in strength between the cylinders and the bricks. The remaining variability is likely to be due to the difference in density and moisture contents.

Effect of curing temperature. The effect of elevated temperatures on the compressive strength was evaluated. In all test mixes apart from the unstabilised specimens, curing the specimens for two days at 105°C increased the compressive strength. This decrease in strength for the unstabilised specimens is unexpected, but is statistically insignificant, and can be accounted for by the variability

of testing. None of the stabilised specimens cured at 20°C statistically showed an improvement in strength compared to the unstablised specimens, with the specimens containing 3% sodium hydroxide and 15% sodium silicate giving a significant compressive strength reduction.

Only the specimens with sodium silicate are statistically significantly different in strength due to curing, with a p-value of a two-tailed t test of less than 0.05. The maximum strength achieved was 10.4N/mm^2 by the specimens containing 5% sodium hydroxide and 20% sodium silicate. This is five times the strength of the equivalent specimens that were unheated.

It is clear from Fig. 4 that the curing temperature is critical with respect to wet compressive strength. Only the samples that were cured at 105°C could be measured. The compressive strengths achieved are negligible compared to the initial 1N/mm^2 minimum strength criterion.

Effect of Geopolymerisation. The contributing factors to a successful stabilisation utilising the geopolymerisation mechanism can be evaluated using the compressive strengths. Since the samples cured at room temperature disintegrated under water, only the compressive strength from the specimens heated to 105°C will be considered.

There is an 80% strength increase with the addition of either 3 or 5% sodium hydroxide compared to the unstablised sample, but there is negligible difference between the quantities of additives. A significant increase in strength is achieved with the addition of sodium silicate. The addition of 15% sodium silicate to the 3 and 5% sodium hydroxide solutions resulted in a 5.3 and 4. 4N/mm^2 strength increase.

There were only three samples of specimens tested with 5% sodium hydroxide and varying amounts of sodium silicate solution. It is clear from Fig. 3 that increasing the amount of sodium silicate solution increases the compressive strength. Analysing all of the specimens indicate that the addition of sodium silicate is the greatest contributor after the curing temperature to strength gain. This is evident by the 1% sodium hydroxide with 4% sodium silicate solution.

The importance of sodium silicate to the dry compressive strength and therefore geopolymeristaion is not seen in the wet compressive strengths as in Fig. 4. Only the 3% sodium hydroxide and 15% sodium silicate gave a greater wet compressive strength compared to the specimens without sodium silicate. As discussed above the absolute values of wet compressive strength is negligible due to the significant variation in measured strength.

Summary and Conclusions

The mechanism involved in geopolymer formation can be used to increase the strength of this soil. The strength increase is dependent on several variables, but without understanding the effect of these variables, the compressive strength can significantly decrease.

This study has indicated that curing temperature is the most influential variable for this particular soil. This is seen in all the specimens with additives. Only the specimens cured at the elevated temperature were able to be tested following 24 hours of full submersion in water. There is clearly scope for further research in the optimisation of curing regime, varying both temperature and time to achieve further strength gains.

There is further work to be done on the optimum additive quantities. It has been shown that using sodium silicate is significant with respect to strength gain. Stabilising using 1% sodium hydroxide and sodium silicate indicates that strength gains can be achieved with minimal environmental impact.

The samples were created by compacting soil into small cylinders. Due to the small nature of the samples and inherent variability then the suitability to represent full scale bricks needs to be considered. The provisional work presented here indicated that there was a reduction of approximately 15% in density but is significantly effected by the difference in aspect ratios. The cylinders represent an average strength of only 50% of that compared to full scale bricks.

This work has shown that addition of alkaline activators for stabilising against elevated moisture contents has been ineffective for this soil, indicating geopolymerisation was not successful. Examples in the literature have shown successful geopolymerisation may have been achieved with another soil or a different process, but as noted in the literature it is not possible to quantitatively predict whether a particular soil is suitable for geopolymerisation. A different soil will have a different mineralogical and chemical composition compared to the one studied here and this change in molar ratios may be sufficient to lead to a successful stabilisation through geopolymerisation.

An inherent characteristic of brick soil is the particle size distribution of the soil that is composed of majority of fines. The literature generally did not discuss the effect of particle size distribution, which is known from other earth construction techniques and traditional concrete design to have a significant effect on strength. The process of industrial extrusion is fairly limited with respect to the scope of change and may not provide suitable conditions for the different geopolymerisation stages. Further work on brick soils that could include the addition of calcined material such as metakaolin may lead to a successful geolpolymeristaion and stabilisation. This could then be investigated with the extrusion process on a small or large scale

This work has shown than there is significant potential for strength to be increased with geopolymers. The geopolymeristaion mechanism was shown to be ineffective at providing sufficient strength under fully saturated conditions for this soil, but this may not be the case for all soils.

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